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# Testing Geothermal-Well Cements: High Temperature, High Pressure, and Fluid Handling Facility

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Washington, DC 20234

October 1979

Interim Report

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Prepared for

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**U.S. DEPARTMENT OF COMMERCE, Philip M. Klutznick, *Secretary***

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## Abstract

Construction of a high temperature, high pressure, and fluid handling facility, which includes four pressure vessels of Hastelloy alloy C, has been completed. The facility allows set cements to be exposed to simulated geothermal fluids at pressures up to 60 MPa (8700 psi) and at temperatures up to 400 °C. Two of these pressure vessels are equipped for measuring either the shear-bond strength of the cement-steel interface or the cement permeability to water at elevated temperatures and pressures. In addition, there is a fifth pressure vessel of stainless steel 316 which can be used for set-curing cements in molds under water at pressures up to 21 MPa (3000 psi) and at temperatures up to 340 °C.

## Introduction

The facility which is described below was built as part of the development of a laboratory testing technique for evaluation of certain physical properties of set cements which are candidates for use in finishing geothermal wells. Selection of a cement for cementing a steel pipe in a well is particularly important to avoid well failure due to cement degradation. Long term operation of a well is necessary because a major cost of producing geothermal energy is in the drilling of the well. To provide a broad base for identifying improved cements, new formulations of cements have been requested from a number of sources. The laboratory testing technique will provide data on these new cements after they have been set in molds and then exposed to simulated geothermal conditions for extended periods. Properties to be measured include shear-bond strength of the cement-steel interface, permeability to water,

splitting tensile strength, and compressive strength. Having this data, governmental and industrial users can assess these cements as to whether they satisfy certain requirements prior to costly field tests.

The practice for testing geothermal-well cements is described in detail elsewhere [1]. The present report describes the high pressure, high temperature, and fluid handling systems that have recently been installed. Also included is the tentative arrangement for measuring shear-bond strength and permeability at elevated temperatures and pressures.

### High Pressure System

A schematic sketch of the high pressure system is shown in figure 1. It includes four high pressure vessels of Hastelloy alloy C-276. Each of these pressure vessels has an inside diameter of 50 mm, an inside length of 250 mm, and three ports, two for the entrance and discharge of fluids, and one for a sheathed thermocouple (not shown in figure 1). Each pressure vessel is mounted in a separate, opened back module (76 cm, 76 cm, 122 cm high). Steel panels (3 mm thick) which are bolted to steel framing channel (4.1 cm square) form the sides of these four modules. Fluid flow to the pressure vessels is controlled by high pressure valves of Hastelloy alloy C-276 which are mounted behind and whose stems protrude through the front panels of the modules. Pressure is indicated by bourdon gauges which have monel coils and sockets, and over-pressure is limited by safety rupture discs of Hastelloy alloy C. High pressure monel tubing (2.1 mm inside diameter) directly joins the vessels and their accessories through coned and threaded high pressure fittings. These pressure vessels will be used to expose set cements to simulated geothermal fluids at pressures up to 60 MPa (8700 psi).

A fifth pressure vessel of stainless steel 316 (127 mm inside diameter, 300 mm inside length) is mounted on a nearby stand and is equipped with a packed stirrer, an internal cooling coil, a bourdon pressure gauge, and a safety rupture disc. This vessel will be used to set-cure cements in molds under water at pressures up to 21 MPa (3000 psi).

A high pressure metering pump provides pressures up to 69 MPa (10,000 psi), or alternatively, a source of compressed nitrogen, up to 40 MPa (5800 psi). This pump delivers a fluid flow (adjustable up to 13 ml/min) through a Hastelloy alloy C-4 pump head. The output of the pump can be maintained at a constant pressure since it is powered by an air motor that drives a geared plunger against a tantalum diaphragm in the pump head.

A special feature of the high pressure system is the pair of capacitance-sensing, pressure transducers of stainless steel 17-4 PH, which will be operated at room temperature. These transducers respond linearly within  $\pm 0.1$  percent over the pressure range between zero and 69 MPa (10,000 psi) and can be read to a precision within 3 kPa (0.4 psi). They will be used for in situ measurements of permeability and shear-bond strength at elevated temperatures and pressures.

Hastelloy alloy C and monel were deliberately chosen for fabrication of most of the high pressure system in order to avoid generalized surface corrosion and pitting stress corrosion which many metals incur upon exposure to salt solutions, especially at high temperatures. Unfortunately, neither Hastelloy alloy C nor monel was easily available for fabrication of the pressure transducers. Stainless steel 17-4 PH generally has good resistance to pitting stress corrosion at room temperature, and possible corrosion can be limited by the use of oil to separate it from the salt solutions.

## High Temperature System

Five tube furnaces, which are nichrome wire-wound, have been vertically mounted to heat the high pressure vessels individually. One furnace (159 mm inside diameter, 305 mm height) has opened ends and a single heater-zone; it annularly surrounds only the body of the stainless steel pressure vessel while the entire cap and bottom end of this vessel are exposed to room air. This situation poses no serious problem as long as the contents of the vessel are stirred.

The other four furnaces (102 mm inside diameter, 635 mm height) are the split-tube type; each of these has two heater-zones and thermally insulated end plugs. During operation, a split-tube furnace completely encloses a Hastelloy pressure vessel, which is supported upright on a thin-walled stainless steel tube (76 mm diameter) that is packed inside with thermal insulation. The main heater-zone (305 mm length) of a split-tube furnace surrounds the vessel body while the other heater-zone (76 mm length) surrounds the vessel cap. The power to the cap-zone is adjustable as a manually variable proportion of that to the body-zone. These Hastelloy vessels can be cooled about 3 °C/min by compressed-air flow through the furnace interior.

Each of the five furnaces is electrically powered (208 V AC, 3150 W) by a separate temperature controller. The difference between thermocouple input and set point signals is conditioned by proportional mode of control with automatic reset and automatic rate to drive time-based triac output. When the furnaces commence heating the pressure vessels from room temperature, full power provides a heating rate of about 3 °C/min. The controllers maintain a final operating temperature within  $\pm 1$  °C. The maximum operating temperature is 340 °C for the stainless steel vessel and 400 °C for the Hastelloy vessels although the furnaces are capable of much higher temperatures.

Chromel-alumel thermocouples are used to indicate temperature on a digital voltmeter which is calibrated to read within  $\pm 1$  °C. Each Hastelloy pressure vessel is equipped with 3 thermocouples: one junction being located about halfway down along the inside vessel wall; another, around the middle of the outside of the vessel cap; and a third, around the middle of the outside of the vessel body. The inside thermocouple is sheathed in a Hastelloy tube (1.6 mm outside diameter) while the outside thermocouples are sheathed in stainless steel tubes of the same size. The stainless steel pressure vessel is equipped with one thermocouple which is inserted down a thermo-well (3 mm inside diameter) inside the vessel.

#### Fluid Handling System

Three stainless steel cylinders serve as reservoirs for fluids of differing compositions. As figure 1 shows, manipulation of stainless steel valves can direct these fluids through a common manifold to any of the high pressure vessels. Besides distilled water, the fluids we plan to use are light and heavy concentrations of mixed salts ( $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{CaCl}_2$ , and  $\text{NaHCO}_3$ ) in aqueous solutions [1]. A mechanical vacuum pump is attached to evacuate the reservoir cylinders as well as the high pressure vessels before introduction of the fluids. Either salt solution can be equilibrated with a given partial pressure of carbon dioxide to prepare a simulated geothermal fluid, whether it is in its reservoir or in a high pressure vessel.

A pH meter is available to monitor the acidity of fluids. A glass measuring electrode and a reference electrode are mounted in a stainless steel chamber in line between the reservoir cylinders and the high

pressure metering pump. These electrodes can be used to make pH measurements of the buffered carbonic acid solutions at pressures up to 1 MPa (145 psi).

#### Arrangement for Measuring Permeability and Shear-Bond Strength

As figure 1 shows, either of two Hastelloy pressure vessels can be connected to the pair of pressure transducers for conducting a test of cement permeability to water or a test of the shear-bond strength of the cement-steel interface. These Hastelloy vessels will be used for in situ measurements at elevated temperatures and pressures. Another vessel of stainless steel, not heretofore discussed, can be connected to one pressure transducer and will be used only for permeability measurements at room pressure and room temperature.

Using the appropriate practice [1], specimens of cement for either test are set-cured in a mold as shown in figure 2. However, those specimens which are designated for the shear-bond strength test are molded to encase a smooth steel rod (10 mm diameter, 28 mm length) which is centered by recessed cover plates at both ends of the mold. A commercially available one-inch-pipe hex nipple of stainless steel 316 (22 mm inside diameter, 57 mm length) was modified to serve as the mold. The nipple was sawed in half, and the inside diameter of the resulting pieces was tapered in a conical fashion such that the opening at the pipe threaded end is about 5 percent larger than the opening at the hex end. Thus, when pressure is applied to the specimen at the pipe threaded end, the specimen tends to make a better seal with the mold.

When measurements are to be made at elevated temperatures and pressures, the mold of set cement is installed within a Hastelloy pressure vessel to make two concentric chambers. The conical plug or ring of set cement serves as the weakest partition between these two chambers. The threaded end of the mold is fitted with teflon pipe sealant into a hex reducing coupling which is attached to the underside of the head of the pressure vessel by stainless steel tubing (4.6 mm inside diameter). The coupling assembly is installed inside the pressure vessel, and valves are opened to make the two chambers common initially. After room air is evacuated, the vessel is filled with distilled water and brought to the ambient temperature and pressure of the measurement, using a furnace and high pressure metering pump as needed. Prior to conducting a test, the two chambers are closed off from each other except for the cement partition. While one chamber is isolated, a pressure difference is applied across the cement specimen.

The details for calculation of permeability and shear-bond strength are given elsewhere [1]. In brief, the permeability is calculated from the decline of the pressure difference as a function of time after both chambers have been isolated. The shear-bond strength, however, is calculated from the maximum pressure difference which is required to displace the smooth steel rod.

#### Reference

- [1] R. F. Krause, Jr. and E. R. Fuller, Jr., "Testing Geothermal-Well Cements: Standard Practice," NBSIR 80-2099-2, National Bureau of Standards, Washington, D.C., Interim Report, July 1979.

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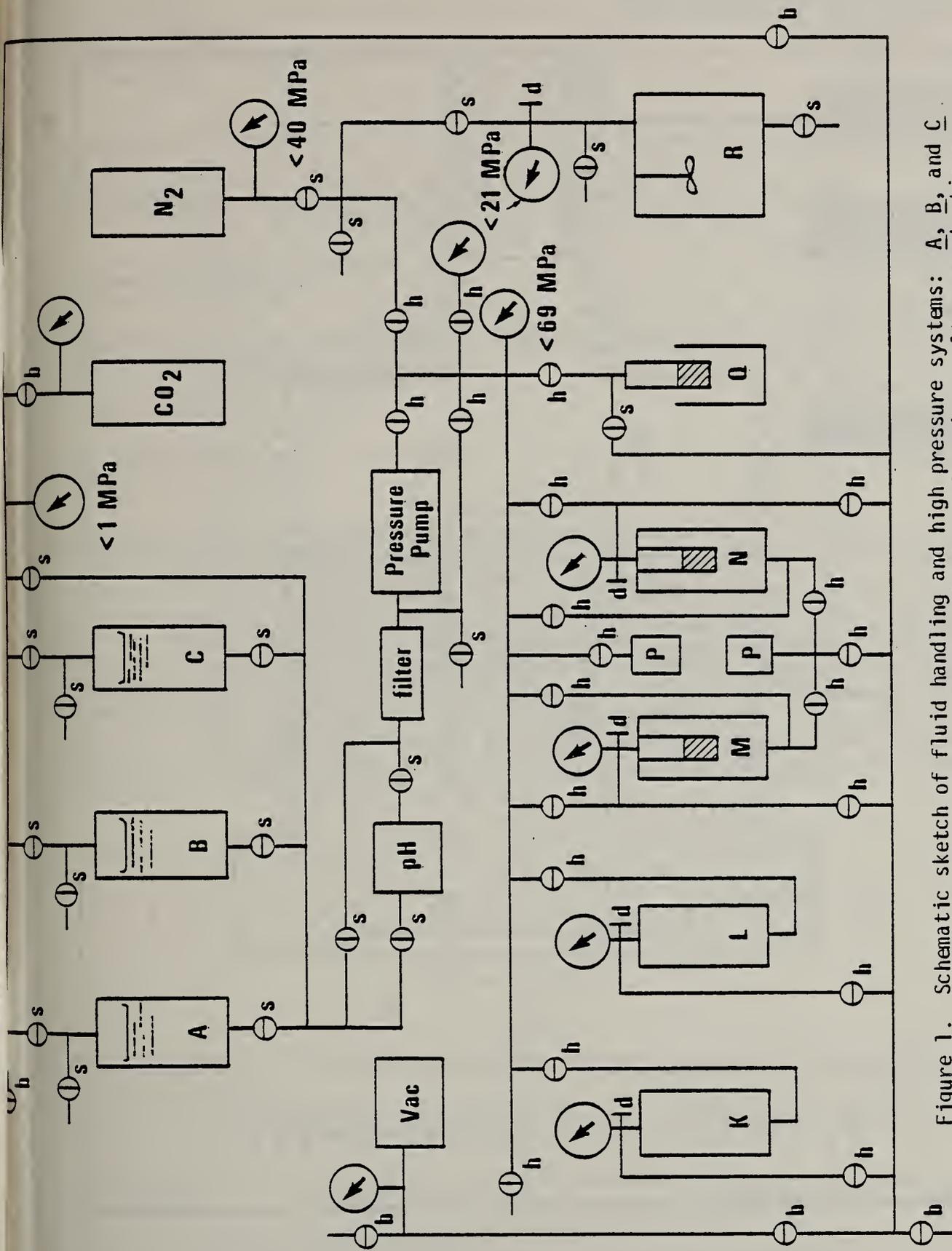


Figure 1. Schematic sketch of fluid handling and high pressure systems: A, B, and C stainless steel 304 reservoirs for aqueous solutions of various salt concentrations; K, L, M, and N Hastelloy alloy C276 pressure vessels (<math>< 69 \text{ MPa}</math>, <math>< 400 \text{ }^\circ\text{C}</math>); P capacitance-sensing pressure transducers; Q cement specimen holder for permeability test (<math>0.1 \text{ MPa}</math>, <math>25 \text{ }^\circ\text{C}</math>); R packed stirrer, stainless steel 316 pressure vessel (<math>< 21 \text{ MPa}</math>, <math>< 300 \text{ }^\circ\text{C}</math>); b brass valves to isolate high pressure monel tubing; and s stainless steel 316 valves to isolate stainless steel 304 tubing.

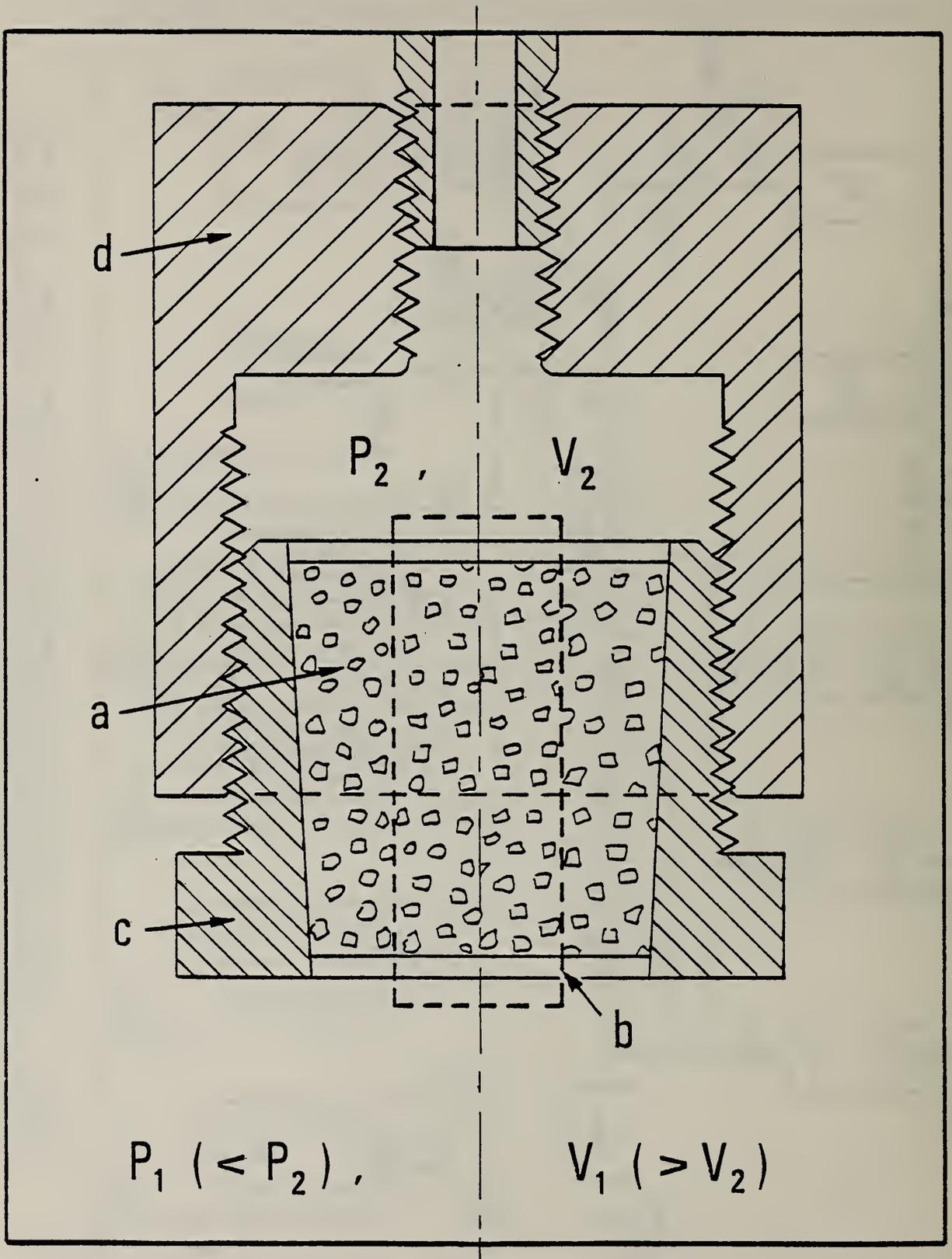


Figure 2. Cement specimen mold and holder for permeability and shear-bond strength tests: **a** specimen of set cement (23 mm lower outside diameter, 24 mm upper outside diameter, 25 mm height); **b** smooth steel rod (10 mm outside diameter, 28 mm length) installed only for shear-bond strength test; **c** stainless steel 316 mold for cement specimen; and **d** stainless steel 316 hex reducing coupling (1 inch NPT to 1/4 inch NPT).

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